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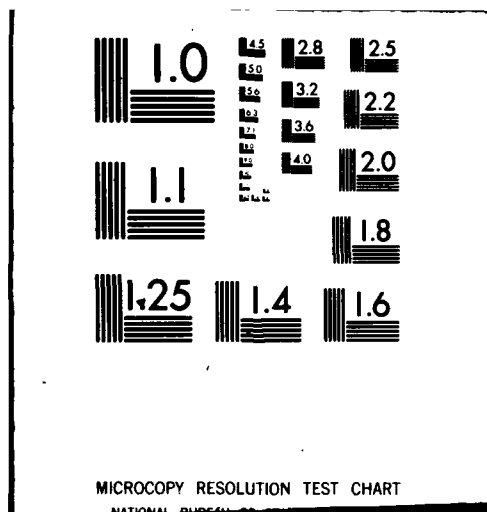
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applications, with the major emphasis placed on the thermal tempering process, as related to glass containers. Thermal tempering methods, apparatus used and the results of actual tests performed are discussed in detail.

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INTRODUCTION

Glass is always considered to be a weak material, exhibiting poor structural qualities. This image, however, is far from true, since glass is inherently very strong. Flawless glass fiber has been found to have tensile strengths over 6.9 GPa (1 million psi). This potential strength coupled with extremely high compressive strengths make glass a desirable engineering material.

The actual strength of a common glass article or plate, because of surface imperfection or flaws, is approximately 5.5 MPa (8000 psi), a reduction in maximum strength by three orders of magnitude. Table 1 provides an interesting comparison of the strength of glass in its many forms.

Fracture of glass nearly always occurs in tension at or near the surface, originating at a surface flaw. Surface conditions therefore are the key to improving strengths if the glass is to be used in any useful structural capacity. Many methods have been found to strengthen glass such as altering the surface, thermal prestressing or surface coating techniques.

Although the techniques of strengthening glass and ceramics are not new, many designers and engineers are unfamiliar with the versatility of this unique family of engineering materials. Glass in the form of plates and simple shapes is not difficult to strengthen, either individually or in mass production. Strengthened glass not only has a high strength, as the name implies, but is reproducible and resistant to extremely adverse environment.

Some of the more practical methods for strengthening commercial glass items can be listed as follows:

1. Acid polishing: etching the glass surface to minimize surface flaws.
2. Chemically tempering: changing the surface composition which will produce a compressive state.
3. Thermally tempering: quick cooling of the heated glass to induce a compressive stress near the surface.
4. High temperature lamination: a surface layer of glass of a differing composition.
5. Thin films of organic materials: films applied to the surface of the glass will increase the strength.

Table 1. Strength values for glass

Untreated glass

Theoretical ¹	6.9-27.6 GPa (1,000,000,4,000,000 psi)
Commercially available fibers	1.72 GPa (250,000 psi)
Fibers in plastic	1.06 GPa (150,000 psi)
Pressed ware	55 MPa (8,000 psi)
Bulk glass design strength	3.45-10.3 MPa (500-1500 psi)

Tempered glass 106-138 MPa (15,000-20,000 psi)

Chemically strengthened 0.69 GPa (100,000 psi)

¹Based on differences in assumptions.

The literature abounds with many variations of each of the above techniques and the reader if interested should refer to the references in this report. The strengthening process selected will generally be a function of the glass composition configuration and the thermal and structural environments which the glass will experience.

This report will discuss chemical and thermal tempering with the major emphasis on the thermal tempering process. An examination will be made of the techniques, equipment and the actual tests conducted in evaluating this process.

DISCUSSION

Chemical Strengthening by Ion Exchange Techniques

There are two basic ion-exchange processes which can be used to strengthen glass. The first involves the exchange of larger cations in the glass for lithium ions. This exchange takes place when the glass is placed in a bath of molten lithium salts at temperatures above the strain point of the glass. At these temperatures the glass surface is relaxed and lithium ions are accepted into the structure. Since the coefficient of thermal expansion of lithium glass is less than the interior glass, compressively stressed surface results on cooling.

A second process is a low temperature method employing a different principle than the method previously discussed. The glass article is treated in molten potassium nitrate at a temperature below its strain point. Exchange occurs between the sodium ions in the surface of the glass and the larger potassium ions in the salt bath. Surface compression is a result of the larger ions expanding the surface skin of the glass.

Thermal Tempering

Tempering glass is a heat-treating process, sometimes called "chilling" or "thermal toughening", which can be applied to certain types of glassware, primarily to increase the mechanical strength. The tempering process is the opposite extreme to annealing glass. Annealing consists of heating the glass to a temperature near the softening point, to remove any strain, and then cooling slowly over a period of hours to prevent internal strains from redeveloping. When tempering, the glass is heated to a similar temperature, then rapidly cooled by air blasts or dropping the article into oil fused, inorganic salts. The object is to induce a system of permanent stress into the glass with compression on the exterior surfaces balanced by an internal tension.

When the glass article is removed from the heating furnaces, and the surfaces are quickly chilled, the exterior layers quickly become contracted and rigid while the interior is still fluid and expanded. As the interior cools, it tries to contract; but it is restrained by the rigid envelope. As the glass temperatures approach equilibrium, the stresses at the surface become highly compressive and are resisted by tensile stresses within the interior of the glass body.

For a tempered plate, the stress distribution across the section may be represented by parabola (A, fig 1). The exact shape of this curve depends upon the temperature range over which the rapid chilling takes place, the rate of chilling, the geometrical shape of the glass section, and the physical properties of the particular glass composition used. In order to maintain a satisfactory stress pattern in glass articles of certain geometric shapes, special tempering techniques must be carefully worked out; certain configurations may be found to be entirely unsuitable for the tempering process (table 2).

To date there are no companies in the United States that are engaged in the production of thermally-tempered containers. In the future, however, at least three companies are expected to begin producing bottles which will meet government safety requirements. The new term used to describe the tempering process is "Controlled Annealing." In Europe, on the other hand, there are some 50 to 60 companies producing tempered ware. Figure 2 shows a commercial French-made tempered glass bowl and tumbler. The stresses in these items are extremely high as evidenced by the way the glass fractures when the compressive layer is physically disturbed, and the color pattern observed under the polariscope. This particular bowl bounced on concrete when dropped from a six-foot height.

Thermal Tempering Limitations

There are some drawbacks to the thermal tempering process which can limit its application. The configuration is important since certain shaped articles cannot be uniformly stressed during the chilling operation. Figure 3 indicates those shapes which are ideally suited to the process and shows others that will fail or have high uneven stresses.

An additional limitation is the thickness of the glass and the uniformity of the glass thickness. Although glass articles with thicknesses of 1.5 mm (.06 inches) have been successfully tempered it is more advisable to temper items greater than 5 mm (.2 inches). The risk of distortion is greatly enhanced with a thin wall article.

Table 2. Advantages and disadvantages of strengthened glass

	<u>Advantages</u>	<u>Disadvantages</u>
Thermal tempering	<ol style="list-style-type: none"> 1. Inexpensive technique 2. Short time required 3. Thick compression zone 4. Simple equipment 5. Most glass compositions 6. Mass production 	<ol style="list-style-type: none"> 1. Thickness limitations 2. Configuration limitations 3. Chilling must be controlled 4. Possible distortion
Chemical strengthening	<ol style="list-style-type: none"> 1. No thickness limitations 2. Higher strength than thermal tempering 3. No distortion 4. Many possible configurations 	<ol style="list-style-type: none"> 1. Long process time 2. Thin compression zone 3. Special glass compositions 4. Proprietary

Behavior of Tempered Glass

If tempered glass is struck by a sharp object or if the surface is penetrated with a score, a flaw will be introduced to the tensile zone. The strain energies released by the fracture will cause a network of cracks to spread through the interior. This fracture pattern, composed of small cubical bits, is known as "dicing." In general, the higher the degree of temper the finer are the fragments into which it disintegrates. Figure 4 illustrates dicing patterns for various types of glass.

The ability of tempered glass to completely destruct can be readily illustrated by water quenching a thin stream of molten glass and breaking the resulting solidified twisted fiber. A break in the surface anywhere along the fiber will result in a pile of glass dust.

Use of Strengthened Glass

Glass in a strengthened or frangible condition has found many uses in defense as well as nondefense related industries. Taking advantage of the material's unusual behavior a number of interesting and useful applications have been developed. Among these applications are:

1. Safety windows which shatter when accidentally broken.
2. As a shock absorber due to stored energy.
3. Restaurant glass articles that resist breakage.
4. Automobile windows.
5. Pipettes and centrifuge tubes.
6. Deep-submergence vehicles for instrument housing and flotation.

Experimental Thermal Tempering

This report will clearly illustrate the feasibility of thermally tempering glass containers within a laboratory environment, economically, with minimum equipment and a simple source of air for chilling. Obviously this method cannot be successfully used to strengthen all glass configurations, since there are definitive size and shape limitations. The principle, however, can be adapted to a large variety of shapes with proper air adjustments and holding apparatus. The main concern in any chilling operation is that the opposite surfaces of the glass must be cooled evenly to prevent stress imbalance.

The distribution of the cooling air can be tailored to the work, and this can be done by adjustments in advance of actual operation of the tempering apparatus. Examination of the stress patterns produced provides the basis for the next adjustment, and so on until a satisfactory result is obtained.

Tempering Flat Plates

To develop a technique of tempering containers and to satisfy an exploratory program requirement, initial tempering tests were performed on numerous flat plates. A large 6.35 mm (.25 inches) thick soda-lime glass plate was cut into 76 mm x 76 mm (3 x 3 inches) squares with each square subjected to a specific tempering environment. Basically the tests were conducted by heating each plate to a temperature just below the softening point and quenching. The quenching environment spectrum included immersion in silicone oils to mild air chilling.

Figure 5 shows the air chilling apparatus which provided a successful technique for flat plates. The glass plates were heated in a kiln to 650°C (1200°F) which is slightly below the softening point of the glass. After a 15-minute soaking period, the glass was removed from the kiln and air chilled. The glass plates were viewed under a polariscope and definite strain patterns were evident as shown in figure 6.

Following the tempering of flat plates, work was initiated on container shapes. In the initial tempering tests, peanut butter jars were extracted from their molds when hot and then chilled. Successful tempering of over 30 peanut butter jars led to the development of a simplified tempering technique where the bottoms of an assortment of commercial jars were tempered.

Tempering Peanut Butter Jars

The feasibility of using tempered glass as a structural material for an ARRADCOM project was discussed with many companies. Brockway Glass, Brockway, PA was ultimately funded to provide technical help in terms of tempering a small number of jars taken directly from a production line.

The tempering was accomplished at the Brockway, Washington, PA plant by air chilling the container immediately after removal from the forming mold at 760°C (1400°F). Figure 7 shows the tempering apparatus used, and the location of the jar during the chilling operation. When the hot jar is placed in position, it is rotated and heated by means of two gas burners. At 820°C (1500°F) the burners are shut off and air is evenly sprayed on internal and external surfaces. When a

temperature of 40°C (100°F) is reached, the air spray is removed and the jar is allowed to cool to room temperature naturally. The curves in figures 8 and 9 indicate the cooling rates experienced by the jars during the tempering operation. A total of 33 jars were tempered in the manner described above. Table 3 summarizes all the work performed at Brockway on both working days, indicating the various methods attempted.

Fifty-one Brockway peanut butter jars were tempered, 34 of which were successful. The degree of tempering and the sensitivity of the jars are presently unknown; however, viewing the container under polarized light indicates residual stress does exist. Since some jars (10) failed (shattered) some time after tempering, this is an indication that we approached the upper stress limit for these particular containers.

Tempering In-House

Our ultimate objective was to provide a method of tempering a glass container without physically holding the glass or setting the glass in direct contact with a positioning stand. A design was developed which allowed the glass container to be supported on a column of air. The air then serves a dual purpose, that of chilling the inner surface of the container and supporting the container. This method of support prevents surface marks which would normally occur with a holding device and allows full exposure of all surfaces without a restricting base or lip support. Figure 10 shows asbestos-wrapped guide posts which stabilize the glass container on the air column without a restricting base or lip support. To obtain glass containers needed to develop the tempering techniques, an assortment of glass bottles was gathered and the bottom portion cut off.

Figure 11 shows a glass tubing cutter which is used to cut the bottles with a hot wire. The glass is first scored with the tubing cutter then it is placed on the hot wire so that the wire is aligned with the score. When the glass is slowly rotated, the glass cracks along the score. This cutter can accommodate glass up to 19 cm (7½ in.) in diameter.

The procedure for tempering containers involves placing the glass article in an appropriate furnace in a lip-down position (fig 12). The furnace is then heated at a rate shown in figure 13 to a temperature slightly above the strain limit of the glass and approaching the softening point. When this temperature is reached, the glass is allowed to soak five to ten minutes before removal.

Table 3. Summary of results of tempering peanut butter jars

Series ¹	No. of jars	Total no. tempered	Tempering environment
I	29	22	Heat to 800°C (1470°F) air quench
II	4	4	Heat to 800°C (1470°F) air quench (gas on)
III	4	0	Deeper internal cooling nozzle
IV	6	2	Restrict air chill to exterior
v	8	6	Moist air chill
	—	—	
Total	51	34	

¹See curves in figures 8 and 9.

Removing the heated glass is accomplished by hand with an asbestos glove. This is done carefully and quickly with the glass article being positioned lip down on the air nozzle. The upper air nozzle is then positioned directly over the glass item. Quenching is accomplished with a continuous blast of air (or other chilling medium) simultaneously on the inner and outer surfaces (fig 10).

Tests have indicated that a container with a hemispherical base and uniform wall thickness (approximately 5 mm) provides the best design shape to temper. In the manufacture of glass, blow mold techniques tend to produce glass articles with variations in wall thicknesses. It is, therefore, better to consider a glass article produced by a press mold method since the wall will be uniform, making tempering more reliable and consistent.

Results

Twenty-one successful attempts were made to temper commercial, mass-produced glass containers (liquor bottles, pickle jars, etc.). When the compressive layer was penetrated some of the jars burst violently with fine dicing patterns, indicating a high degree of temper.

In addition to tempering commercial glass containers, six hand-blown glass containers were successfully thermally tempered. Although they were inferior to the commercial glass they posed no problem in the tempering cycle. Figure 14 illustrates the stress patterns developed in various glass items as viewed through polarized light.

Effects of Glass Color on Tempering

Through experimentation brown-glass and green-glass containers have been tempered utilizing the same method used to temper transparent glass containers. These experiments showed that colored-glass containers appear to reach higher residual stress levels than transparent-glass containers.

Viewing Stress in Glass

The polariscope is an instrument used to reveal the strain in glass, due to tempering or improper annealing. It consists generally of a light source, a polarizer, a tint plate and an analyzer. The model shown in figure 15 is a typical design used by the glass industry to view glassware, enabling them to discard items exhibiting stresses which are liable to fail.

This polariscope will not provide the degree of strain; it is used to reveal internal stresses due to external forces or temper. The sensitive tint plate, located under the eye piece, enables one to see a color distribution where stresses exist. Color changes will indicate the nature of the stress, that is, tension or compression (fig 16).

Standard strain disc (fig 17) can be used as a comparison to the strain in the glass. Although this does not provide a value of temper it does help establish the level.

Evaluation of the Temper Level

Both chemically strengthened and thermally strengthened glasses have been treated in such a way, although by different processes, that surfaces are in compression, whereas the interiors remain in tension.

The center tensile stress is measured using polarized light and a low power magnification. The stresses in the glass cause a difference in refractive index in different directions, producing a series of light and dark fringes. The number of fringes is directly proportional to the residual stress, although there is some error because of the effect of the compressive layer through which the light passes. These fringes are shown in figure 14. To determine strains in a glass container, ring sections are cut in the form of hollow cylinders, 1 to 2 centimeters in height. The cut edges are then ground and polished, or in lieu of this procedure, immersed in a liquid of equal refracting power to render the surface invisible. The rings are then viewed edgewise under the polarizing microscope, which brings out very clearly the amount and character of the strain.

The stresses can be measured either by counting the number of fringes or by inserting quartz wedges having known retardations, until the retardation caused by the stress has been equalized. This retardation is then used to calculate the stress. By using a thin sample, the number of fringes in the compressive layer is reduced and the compressive stress can be calculated. However, if the temper is at a high level it is impossible to cut out a section to analyze. Another drawback, assuming the temper were low enough to cut a section, is that some structural strains will be removed by sawing out a section. Thus, the polariscope is satisfactory for determining the uniformity of the temper but it gives little indication of the degree.

Destructive testing can determine the temper level by deliberately breaking the glass and examining the distribution and size of the resulting diced pieces. Normally, the higher the temper, the smaller the pieces.

Perhaps one of the best methods of testing a tempered glass article for overall strength is the thermal shock quench test. This test is established by quenching in ice water the heated glass, at a level slightly below the severity which a well-tempered article will withstand. Articles are heated in an air-oven at successively higher temperatures followed by direct quench in an ice bath. These tests can be used for a 100 percent test in production, allowing only those "good" pieces to survive.

New Techniques to Determine Stress in Glass

A new technique for determining the compressive stress in glass has been developed by Pittsburg Plate Glass, Inc. The actual device, called a differential surface refractometer (DSR), is based on the fact that the stress produces a difference in refractive index between light rays polarized parallel and perpendicular to the sample surface. The method is limited, however, to flat surfaces so that even in this instance, measurements could not be made directly on the prototype, but must be made on flat bars having the same tempering treatment.

A new method for determining stresses in glass is through the use of a gas laser. Because the laser beam is extremely intense, the fringes produced by the scattered light can be seen in a direction perpendicular to that of the beam. This method eliminates the effect of changes in the stress along with light path. The stress at any interior point can be determined without interference from other stresses in the sample. Another key advantage of this technique is that its use is not limited to flat pieces; the stresses in any shaped body can be measured.

In summary, the normal photoelastic techniques, when applied in a microscope, seem to be adequate for the measurement of a stress profile. Their limitation is that a bar having the same stress distribution as the large body must be prepared in order to use these techniques. The DSR allows one to measure the surface stresses in any large piece provided that a flat surface can be obtained. The laser technique although not completely perfected, would allow measurements to be made directly on an irregularly shaped glass body.

CONCLUSIONS

The work in this report clearly demonstrates the feasibility of thermally tempering soda-lime glass plates and container shapes by air chilling. Although no specific applications for strengthened glass have been mentioned, it is evident that if a need arises, strengthening can be accomplished in the laboratory.

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Patents, Thermal Tempering of Glass

Patent No. 3,847,580

Inventor - George W. Misson

Incidents of breakage in glass sheets during tempering are reduced by conducting the quench operation in steps of which the first is the most severe and the others are accordingly less severe.

Patent No. 3,907,132

Inventors - Harold A. McMastor, Norman C. Nitschke

A system and apparatus made up of components for treating glass sheet material including a furnace for heating the material, a blasthead or quenching apparatus for cooling the material after it has been heated.

Patent No. 3,841,855

Inventors - William S. Montgomery, Jr., William E. Marceau, William G. Bates

The invention is directed to the heating of glass sheets for tempering by dividing the heating furnace into zones and controlling individually the temperature of each zone in such a manner that the sheets are raised to a substantially uniform temperature in a minimum of time.

Patent No. 3,883,339

Inventors - Edmund R. Michalik, James E. Neely, Jr.

Tempering glass sheets by a cooling process using a sublimable cooling medium and air.

Patent No. 3,929,442

Inventor - James E. Neely, Jr.

This invention relates to tempering glass sheets using the heat of sublimation of a sublimable cooling medium to help cool heated glass sheets sufficiently rapidly to impart partial temper. Preferably, the cooling medium contains carbon dioxide as the primary source of cooling.

Patent No. 3,890,128

Inventors - Richard Melling, Donald Curtis Wright, John Pickup

Application to flat and curved plates for aircraft windshields. The glass is toughened by severely quenching by contact with a quenching medium producing a high rate of heat transfer from the glass.

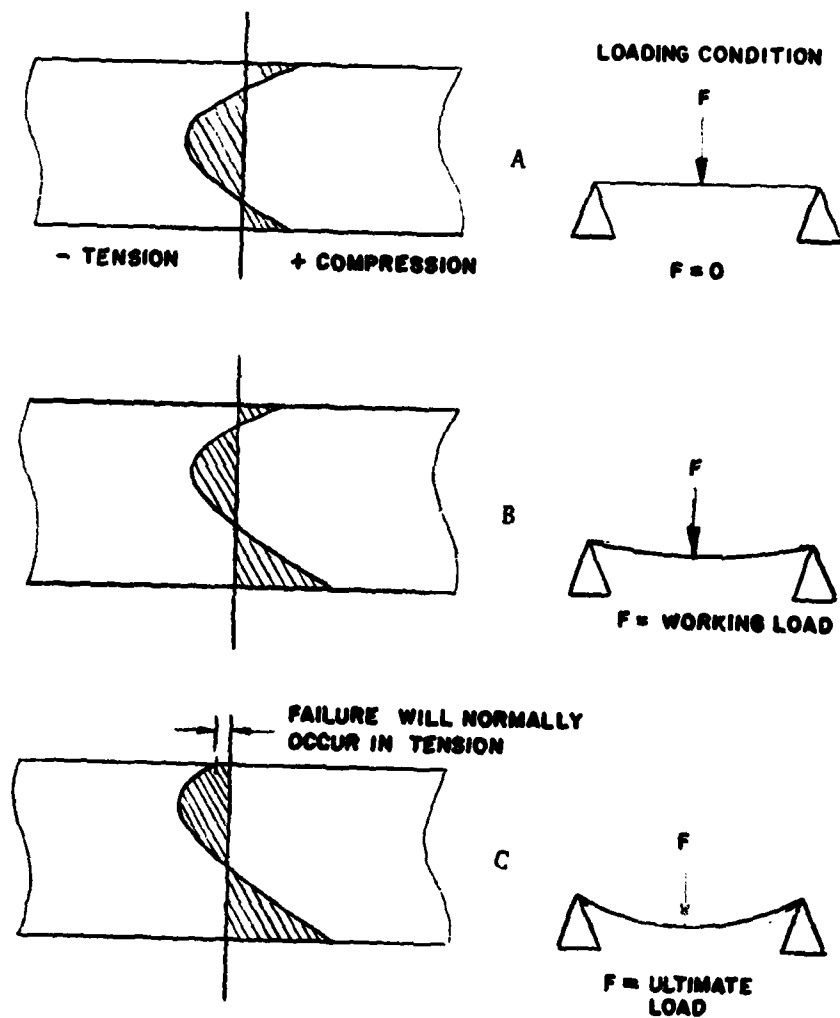


Figure 1. Stress profile in tempered glass at various loading conditions.

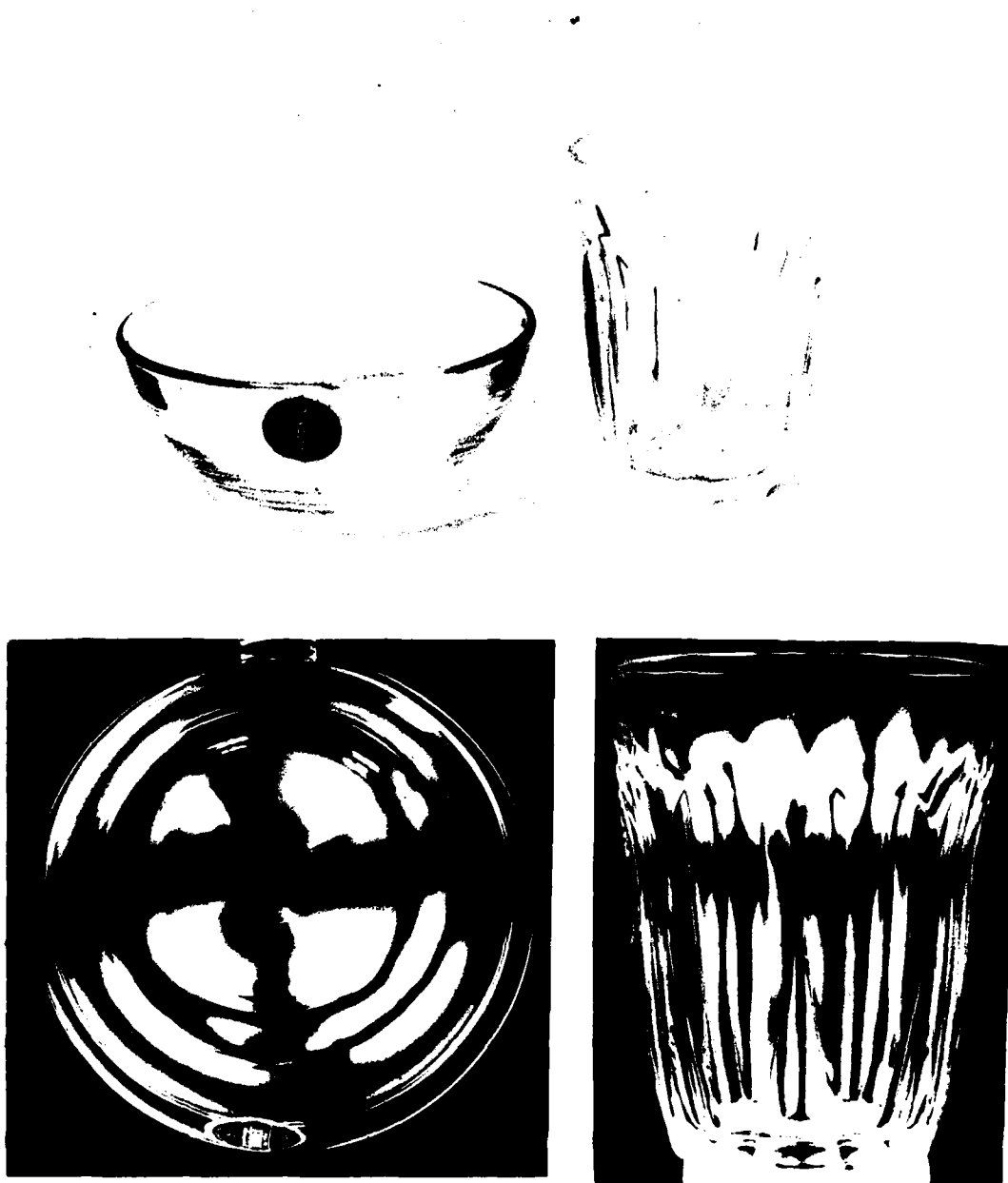
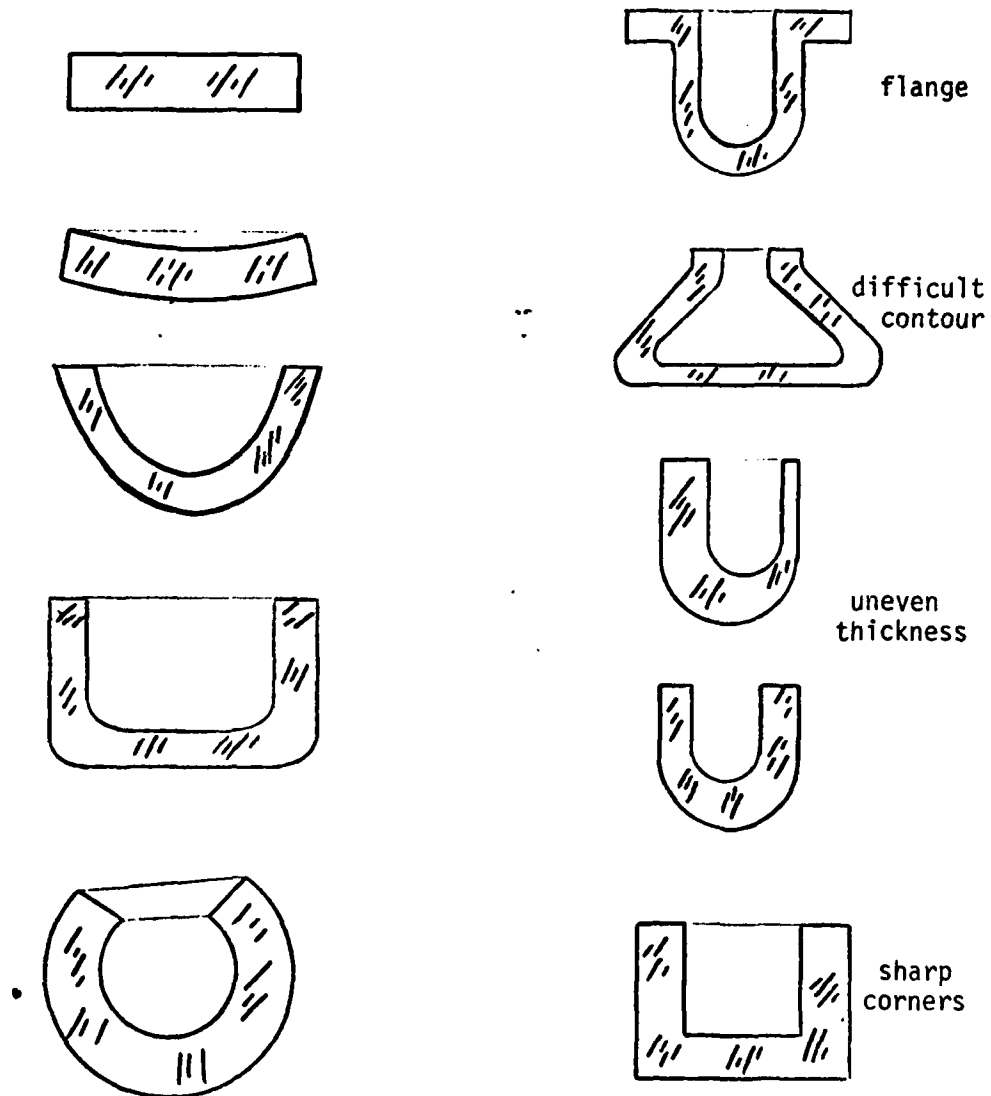


Figure 2. French made "Durelex" tumbler and bowl shown on top with a polariscope view of both (bottom).



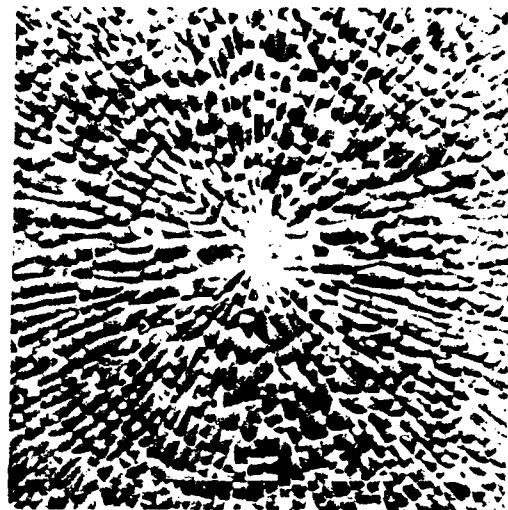
Glass shapes which can be thermally tempered.

Glass shapes difficult if not impossible to thermally temper.

Figure 3. Various glass shapes for tempering.



A. air chilled glass plate



B. chemically strengthened glass



C. commercially tempered bowl

Figure 4. Typical dicing patterns for strengthened glass.

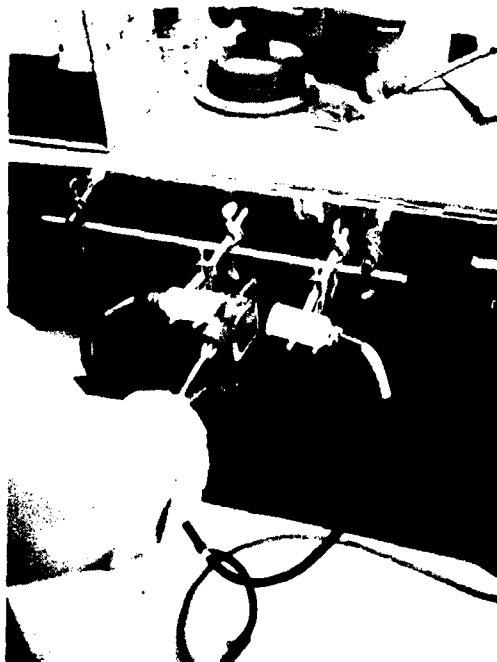
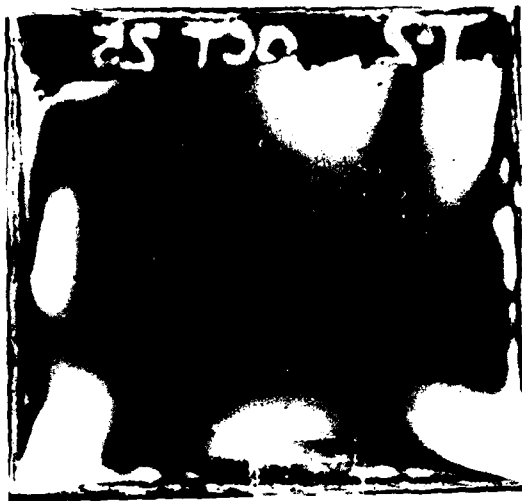


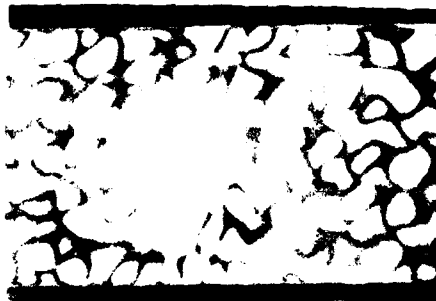
Figure 5. Chilling apparatus for tempering glass plates.



A. 75 x 75 mm plate



B. flat lenses



C. flame tempered

Figure 6. Stress patterns in glass plates viewed under polarized light.



Figure 7. Tempering apparatus used by Brockway Glass to chill peanut butter jars (front and back view).

COOLING CURVES FOR TEMPERED
"SKIPPY" PEANUT BUTTER JARS

BROCKWAY GLASS
WASHINGTON, PA. PLANT
APRIL 25, 1978

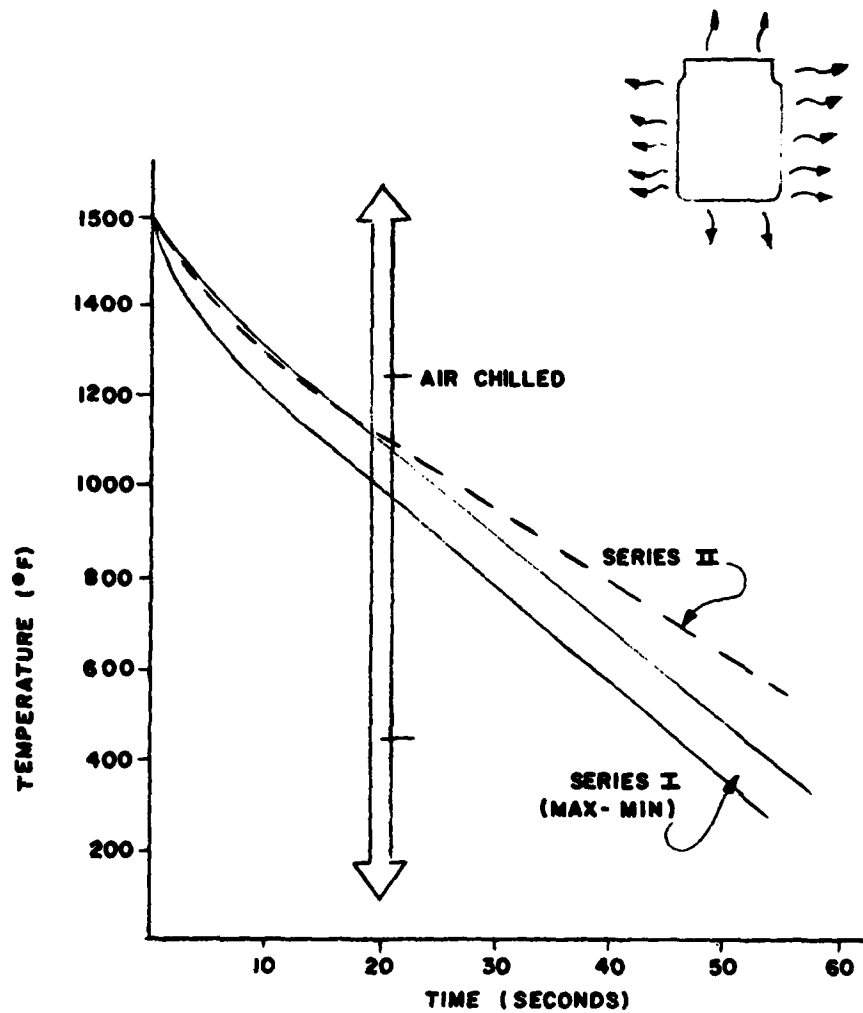


Figure 8. Cooling curves for tempered peanut butter jars
for series I and II.

COOLING CURVES FOR
TEMPERED PEANUT BUTTER JARS

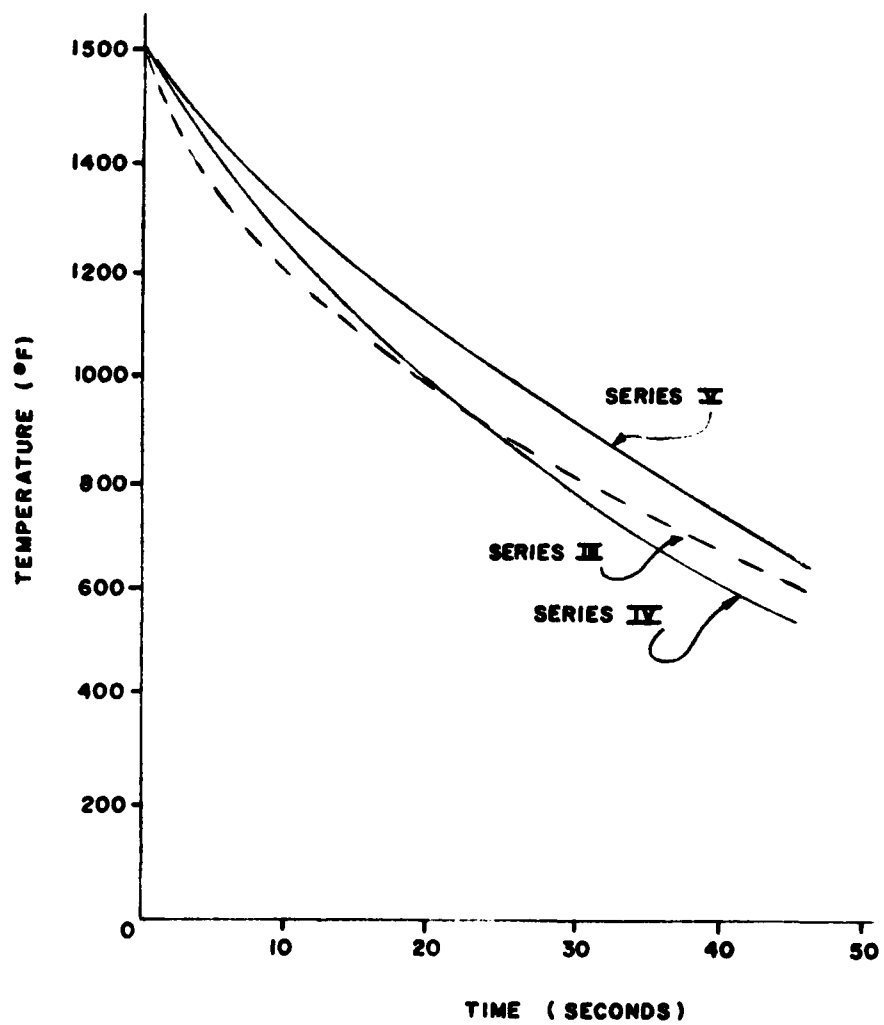


Figure 9. Cooling curves for tempered peanut butter jars for series III, IV, and V.

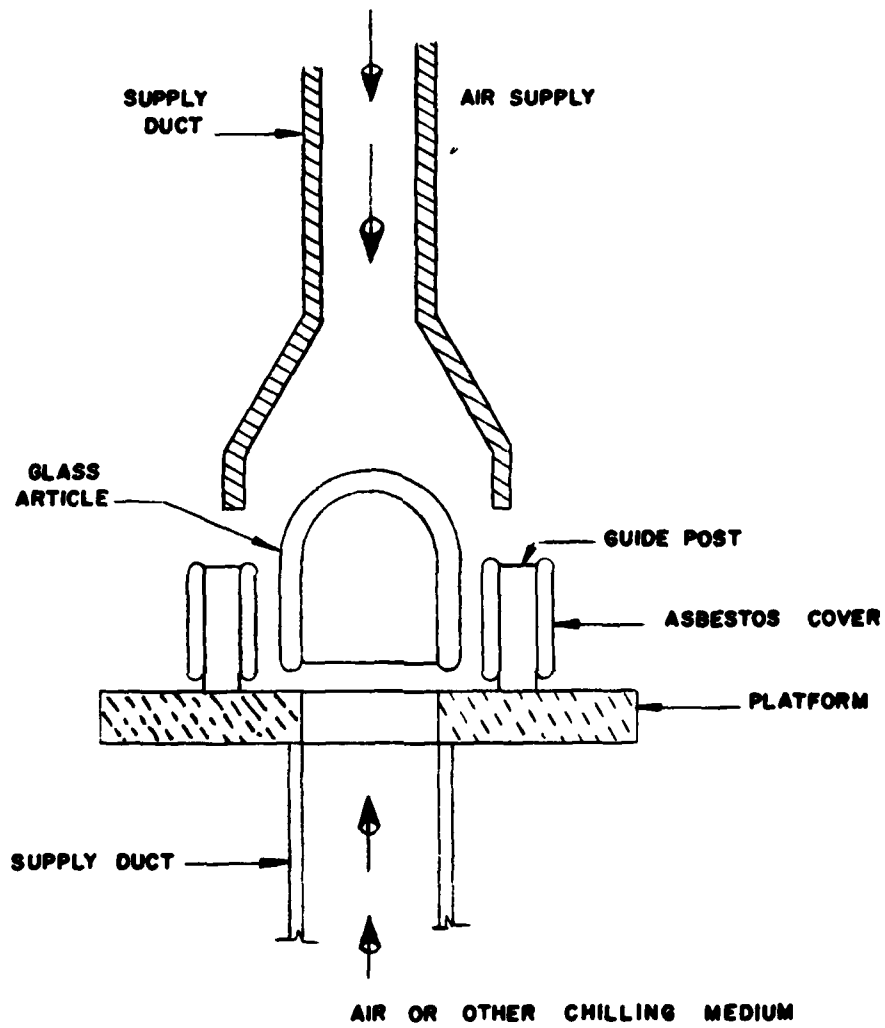


Figure 10. Tempering apparatus.

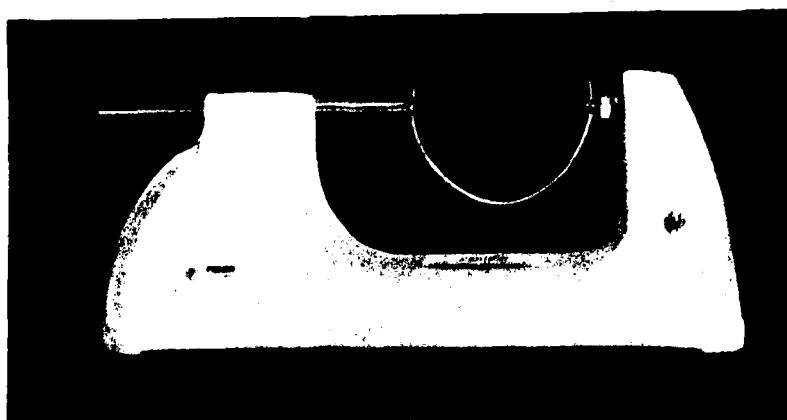
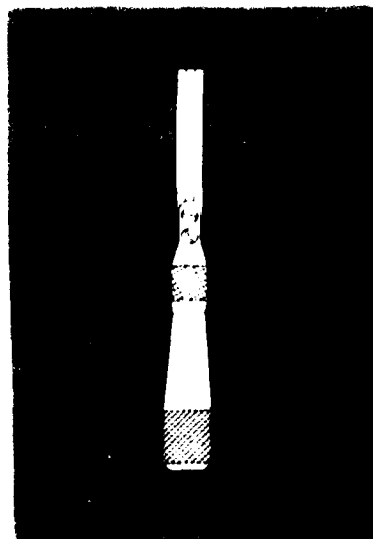


Figure 11. Apparatus used for cutting glass jars and bottles.
(From Fisher Scientific Company catalog.)

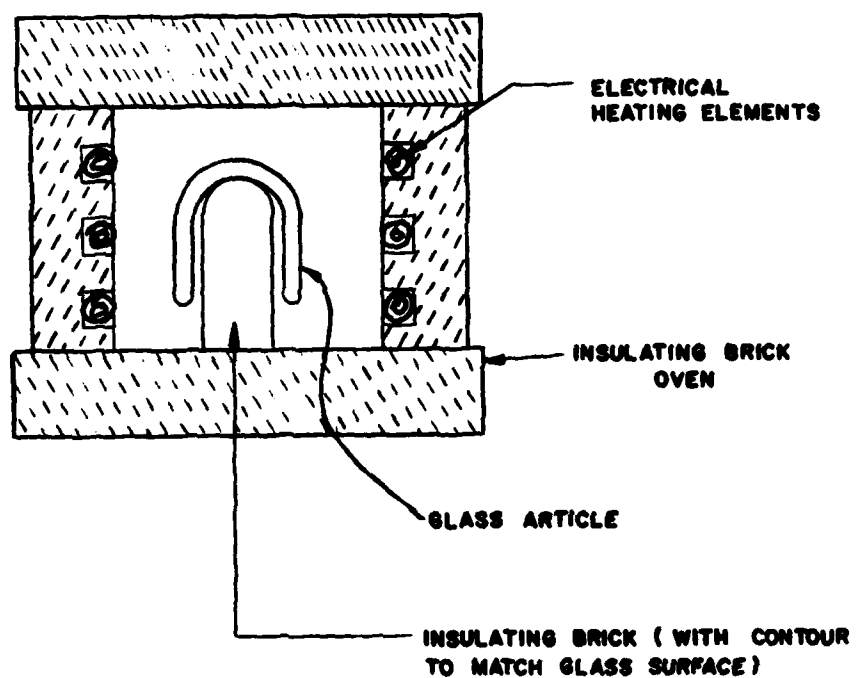


Figure 12. Method of holding glass container while in the kiln.

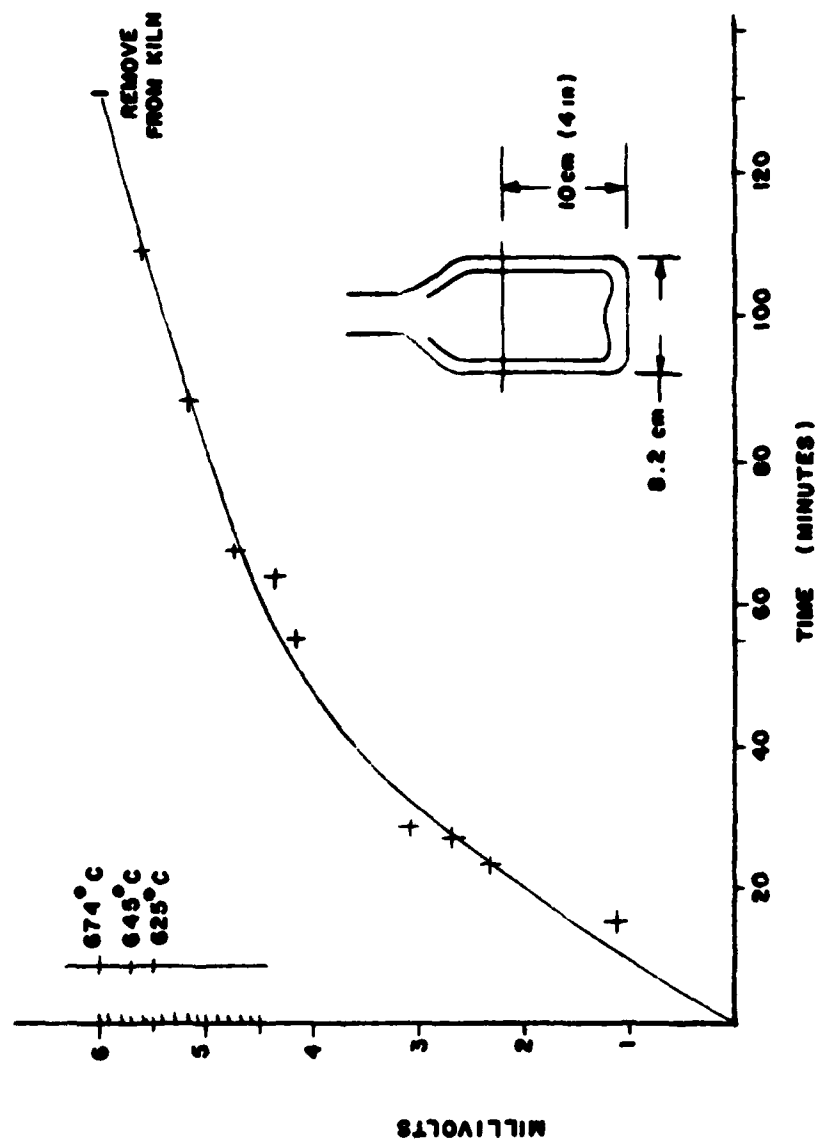


Figure 13. Heating curve used to heat jars and bottles for eventual tempering.

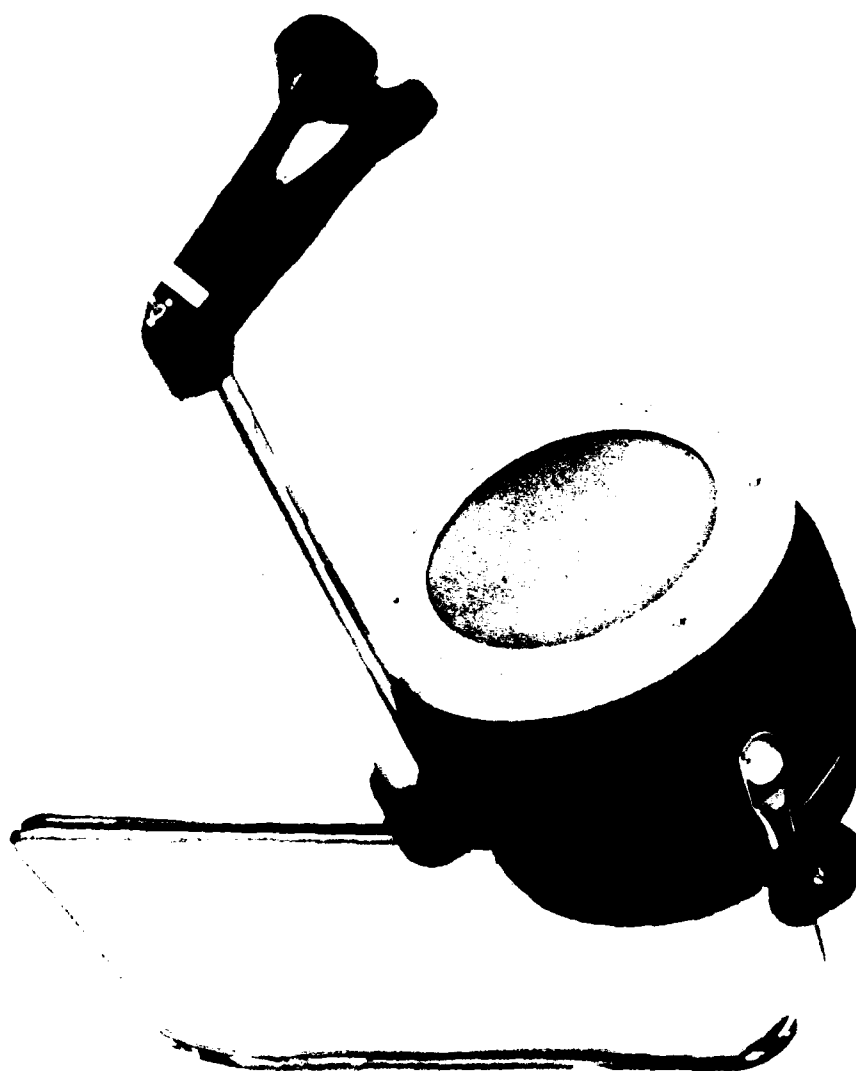


A. Hand-blown glass container (100 cm diameter).



B. Peanut butter jar (1 liter).

Figure 14. Stress patterns developed in glass articles when thermally tempered.



SPECIFICATIONS

Size	36"H x 24"D x 18"W
Polarizer Field	10" Diameter
Weight	42 pounds
Finish	Chrome and Black
Working Distance	16"
Power Required	115V-AC

Figure 15. Commercial polariscope (model 110) manufactured by
Polarizing Instrument Co., Inc.
Irvington-on-Hudson, New York 10533

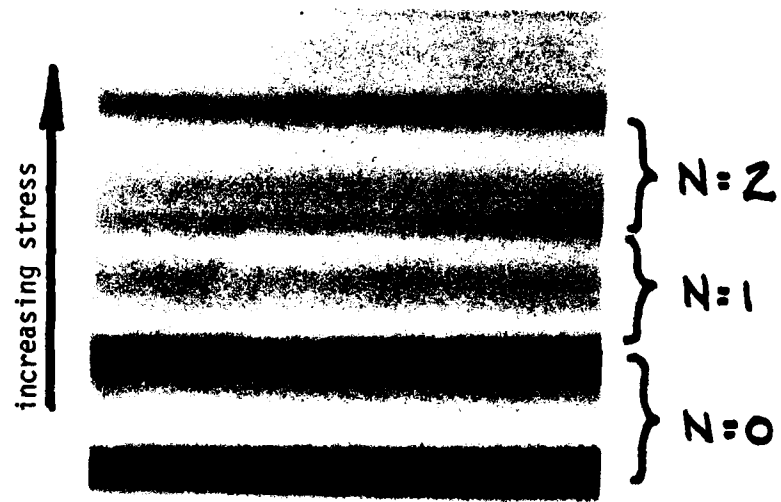


Figure 16. Typical stress pattern as viewed through a polariscope. N represents the fringe order.



Figure 17. Standard strain discs.

APPENDIX A
GLASS CONSULTANTS

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Columbus, OH 43201
Contact: Russell Bennett
(614) 424-7529

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Ceramic Engineering
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Columbus, OH 43210
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Bethlehem, PA 18015
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Brockway Glass
Brockway, PA
(814) 268-3015

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P.O. Drawer 28510
San Antonio, TX 78284
Robert Hoffman
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WASHINGTON, D.C.

U.S. Naval Research Laboratory,
Materials Research Activity
Washington, DC 20375
(202) 545-6700

U.S. Dept. of Commerce
National Bureau of Standards,
Inorganic Glass Section
Washington, DC 20234
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848 Benedum Hall
Pittsburgh, PA 15261
(412) 624-4141

APPENDIX B

CONSTRUCTING A POLARISCOPE

Discussion

Glass when heated to a temperature slightly below the softening point and cooled unevenly or quickly will develop internal stresses. Inducing a stress in glass will not change its appearance. To the naked eye stressed glass looks identical to unstressed glass. However, when viewed through a polariscope stress patterns are quite evident in the stressed glass. One simple form of the polariscope, that can be easily constructed, consists of a wood box with an internal light source, closed at one end with a polaroid filter. When light is transmitted through the polaroid sheet it filters out all light waves except those vibrating in a single plane.

If another polaroid filter is held in front of the box in such a way that it favors light transmitted in a plane 90° away, then the box will appear dark. Since stress glass has the ability to rotate the plane in which the light wave vibrates, stress patterns become visible through the polaroid paper. The stress will appear as patterns of light and shade, while unstressed glass will show no change (fig B-1).

Construction

The polariscope used to study tempered glass was a simply constructed wooden box 35 cm x 20 cm x 25 cm deep made from 1/2 inch plywood. A porcelain socket with a 60 watt frosted bulb was attached to the back of the inner wall. The polarized paper and a light diffuser were fastened to the front wall frame.

For the tempering work this polariscope was very useful in determining a relative degree of stress in the tempered article.

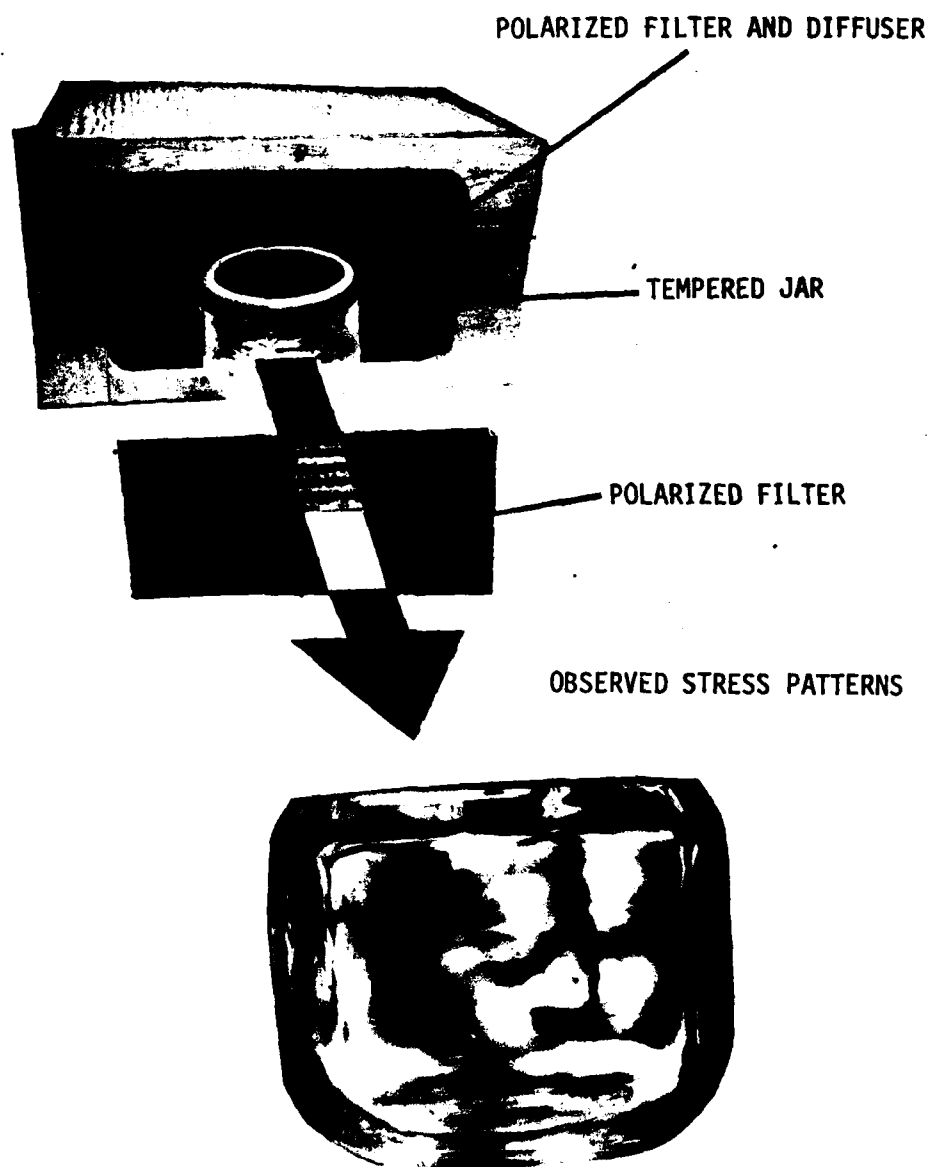


Figure B-1. Polariscope set-up to view stressed glass.

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